RESEARCH ON BORON FILAMENTS AND BORON REINFORCED COMPOSITES

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ABSTRACT

A research program was conducted to explore the potential of boron filament for possible use as the reinforcing phase in composite materials for aerospace structures. The first part of this program was devoted to the evaluation of individual filaments with respect to their mechanical properties. These filaments exhibited an average tensile strength of 350 ksi, somewhat less than that of the S-glass filaments currently used as reinforcement in many composite material applications. Young's modulus of boron filaments averaged 60×10^3 ksi, approximately five times that of glass.

The second part of the research effort was devoted to the evaluation of boron reinforced composites made by winding tapes preimpregnated with Epon 826-Z epoxy resin. Three basic specimen configurations were studied: rings, belts, and cylinders. Several glass specimens of each type were fabricated for comparison with boron specimens. Boron filaments exhibited strengths of about 150 ksi in composite rings as compared with a value of 420 ksi for glass filaments.

The cylinders were fabricated using two different winding patterns - one an orthotropic pattern designed for internal pressure loads and the other, an isotropic arrangement designed for axial compressive loads. Nine elastic constants were determined for four cylinders, one of each material and winding pattern. When the experimental elastic constants were compared with theoretically predicted values, the relative agreement was about the same for boron and glass.

A weight study of a series of large-scale cylinders designed for axial compression and internal or external pressure was performed based upon the elastic constants and tensile strengths obtained from the previously discussed experimental program. The results of this study showed a weight advantage of the boron-epoxy cylinders compared to the glass-epoxy cylinders of about 40 percent for compressive applications; however, a 60-percent weight advantage in favor of the glass-epoxy cylinders resulted for the case of internal pressure loading.

1. INTRODUCTION

During the past few years considerable interest has been aroused regarding the possible use of boron filaments for certain composite material applications where high stiffness and elevated temperature strength are desirable. Three years ago the Langley Research Center of NASA began a research program to explore the potential of boron filaments for use in composite structures. The study was divided into two phases. The first involved the determination of mechanical and physical properties of boron filaments produced by different processes. This investigation of filaments was necessary to provide basic information on mechanical behavior and to determine the best filament type for use in the subsequent composite fabrication program. A total of twelve different commercial types of boron filaments were investigated and the results are reported in reference 1.

The second phase of the research program, not yet complete, consists of design, fabrication, and testing of ring, belt, and cylinder specimens made by winding boron filament in an epoxy-resin matrix. The objectives of the second phase are to compare the performance of the boron epoxy specimens with that of similar glass-epoxy specimens, and to compare experimentally determined elastic constants with theoretically predicted values. This paper presents data on the tensile strength, and tensile and shear modulus of boron filament; the tensile strength, and interlaminar shear strength of boron filament-epoxy rings and belts; and the elastic constants of boron filament-epoxy cylinders.

2. SINGLE FILAMENT INVESTIGATION

2.1 Filament Types

Magnified views of the exterior surface and a polished cross section of a typical boron filament are shown in figure 1. The pebbled appearance of the exterior surface is typical of many vapor-deposited materials. The filament shown in figure 1 was made by a process in which hydrogen and a boron halide react and deposit solid boron on the surface of an incandescent tungsten wire. The temperature of the process is usually sufficiently high to cause reaction between the boron and the substrate so that the final filament contains a core composed of a series of tungsten borides. Boron filament produced in this manner shall be subsequently referred to as halide filament.

Another type of filament, organometallic filament, similar in appearance to the halide filament, is made by pyrolyzing a boron organometallic compound on a substrate wire surface. The process temperature is usually considerably lower than that of the halide method so that less dense substrates such as aluminum and titanium can be used with minimum reaction between boron and substrate.

2.2 Room-Temperature Mechanical Properties

Single filament properties are illustrated in figure 2. Tensile strength is shown on the left where results of several hundred specimens of five different halide filaments and seven different organometallic filaments are averaged. Specimen length was 1 inch between grips. The differences among filaments include the producers, starting compounds used in the boron deposition, and substrate materials. Average halide filament strength is about 350 ksi compared with a value of approximately 100 ksi for organometallic filaments. The low average strengths of the organometallic filaments reflect the fact that these specimens were among the earliest of this type to be produced. The values obtained for organometallic filament in this study probably do not represent the best attainable at the present time.

Modulus values for all filaments tested are illustrated on the right (fig. 2) for 10-inch gage length specimens. Young's moduli of five different halide filaments averaged 60×10^3 ksi compared with a value of 30×10^3 ksi for three organometallic filaments. The lower measured Young's modulus of organometallic filament may be due to the occlusion of low modulus polymeric boron hydrides within the filament during deposition.

The halide filament shear modulus of 18×10^3 ksi was determined by both torsion pendulum and torque-twist techniques reported in reference 2. Values from tests of three different filaments are shown in figure 2.

The magnitude of the scatter among the mechanical property data is quite large. This is especially true for the tensile strength results. Boron filaments are a brittle material. The introduction of internal flaws of varying severity is seemingly unavoidable in the manufacturing process and, therefore, some scatter is to be expected. This amount of scatter, however, was the cause of some concern in the determination of design allowable strengths to be used in the subsequent fabrication of composite structural test specimens. The distribution of tensile failures for 120 tests of a typical halide filament is shown in figure 3. The distribution is based on the number of failures occurring in 10 ksi stress intervals ranging from about 250 ksi to 500 ksi. The average strength is 370 ksi with nearly an equal number of failures occurring above and below this value.

Effect of length on strength. The effect of length on strength is shown in figure 4 where the tensile strength of a halide filament is plotted as a function of specimen length from 1 inch to 240 inches. Similar data for E-glass was taken from the literature (ref. 3) and plotted for comparison. Within the range of values plotted the slopes of the boron and glass curves are nearly equal indicating a similar effect of flaws in the two materials.

2.3 Effect of Elevated Temperature on Strength

The typical effects of temperature on halide filament strength are shown in figure 5. The uppermost pair of curves were obtained from tensile tests conducted in either air or argon at temperatures up to 1750° F. Each specimen was held at temperature for 60 seconds before testing. The lower pair of curves are results of tensile tests conducted at room temperature on filament specimens previously exposed 15 minutes in either air or vacuum (1×10^{-4} torr) at temperatures up to 1500° F.

The curves at the top of the figure are indicative of the elevated temperature load carrying capability of boron filament. Strength remains essentially constant for tests in argon at temperatures up to approximately 800° F. At 1000° F, filament strength has dropped to about half its room-temperature value, and at 1750° F the filament strength is nil. The curve for filaments tested in air falls below that for filaments tested in argon. Apparently this is due to surface oxidation which reduces the effective diameter of the filament.

During fabrication of boron filament reinforced composite structures the filament will be subjected to resin curing cycles, or possibly metal-matrix sintering operations. The effects of elevated temperature exposure on subsequent filament strength under ambient circumstances are of importance. The effect is most severe when the specimens are exposed in air because of surface oxidation. It can be seen that elevated temperature exposure in vacuum reduces the strength of the filament only slightly. If the surfaces of the filaments are shielded from air by the matrix or some other means during an elevated temperature fabrication procedure, the vacuum exposure curve should be a more realistic basis for predicting filament behavior.

3. COMPOSITE MATERIAL INVESTIGATION

3.1 Boron-Epoxy and Glass-Epoxy Composite Rings

Once the properties of individual boron filaments had been reasonably investigated, attention was directed to a study of the behavior of these filaments in a composite material to determine how efficient boron was in comparison to glass in structural configurations.

In order to investigate the tensile strength and interlaminar shear strength of boron filament-epoxy composites, 3-inch-diameter rings, $5\frac{3}{4}$-inch-diameter rings, and belt specimens shown in figure 6 were fabricated from 20 filament tapes preimpregnated with Epon 826-Z resin. The filament used was one of the halide process filaments which had exhibited the best overall properties in the single filament tests. Several similar specimens were fabricated from S-glass filament and the same epoxy resin for comparison.

Room-temperature composite strengths. Room-temperature tensile strengths of the specimens are summarized in figure 7. Filament tensile strength calculated from composite test data is compared with virgin filament strength for both boron and S-glass. The average strength of boron-epoxy rings and belts was about 170 ksi. Average strength of $5\frac{3}{4}$-inch-diameter glass rings was 420 ksi, which was much higher than the boron. Boron ring efficiency expressed as the percentage of virgin filament strength attained by the filament in composite form was approximately 40 percent compared with a value of 60 percent for S-glass rings. A possible reason for this difference could be the inability of the matrix to transfer loads around discontinuities and premature filament failures as efficiently in the case of the boron reinforced rings. The extreme disparity in Young's moduli between the boron and resin would suggest that a much greater length of filament as compared to glass would be required to pick up the load from an adjacent filament at a discontinutiy (ref. 4).

Interlaminar shear strength. The interlaminar shear strength based on the bending of short beams cut from $5\frac{3}{4}$-inch-diameter rings is plotted in figure 8. Average shear strength for 10 boron beams was 8 ksi compared with a value of about 9 ksi for S-glass. The value for glass is within the range generally regarded as acceptable for glass-epoxy composites. Since composite interlaminar shear strength is an indication of the strength of the bond between filaments and matrix, it is believed that the values obtained for boron composite specimens is an indication of reasonably good bonding between boron filaments and the resin system.

Effect of temperature on composite strength. The effect of temperature on the strength of 3-inch-diameter rings is illustrated in figure 9. Specimens were held for 10 minutes at temperature before testing. The strength data are plotted as a percentage of room-temperature strength over a temperature range from -325° F to +400° F. The solid curve for rings shows strength declining gradually from about 125 percent of the room-temperature value at -325° F to approximately 50 percent at 400° F. The rate of strength decay from room temperature to 400° F is less severe in the case of single filaments as shown by the dashed curve. This may be due to resin degradation in the rings as a result of the increased temperature.

3.2 Composite Structures Investigation

Test specimens. Fourteen cylindrical specimens 6 inches long and 3 inches in diameter were machined from domed-end cylindrical bottles 9 inches in length (see fig. 10). Seven bottles were wound with tape composed of 20 halide filaments preimpregnated with Epon 826-Z resin and the remaining seven were wound with 20-end S-glass roving preimpregnated with the same resin. Filament orientation of the cylinders was of two types as shown in figure 10. The orthotropic pattern, consisting of a two-to-one ratio of circumferential to longitudinal reinforcement, was used in four boron and four glass specimens designed for internal pressure loading. The remaining six cylinders, three boron and three glass, were wound with the isotropic pattern - a series of alternating layers of circumferential and opposed helical wraps 30° to the cylindrical axis. The isotropic pattern is optimum for buckling due to axial compressive loads (ref. 5). The actual wrapping patterns deviated from the ideal in each case. In the orthotropic pattern, the longitudinal filaments were wrapped at a 7° angle to clear the mandrel end bosses. In the isotropic pattern, the helical filaments were wrapped at a 33° angle instead of 30° because of an error in setting up the winding machinery.

The four types of bottles from which cylinders were machined are shown in figure 11. An examination of the cross sections of these cylindrical specimens revealed irregularities as shown in the photomicrographs in figure 12. The glass cylinder cross section exhibits a normal appearance for this type of material. The boron cylinder cross section, however, contains large, irregularly shaped voids located at the ends of the cylinder primarily within the layers which traversed the domed ends of the bottle. When all loading tests have been completed, cross sections will be cut from the central portions of the cylinders to determine the extent of voids.

Test program. The types of loads to which the cylinders were subjected, and the elastic constants determined from each type of loading are indicated in figure 13. Each cylinder specimen was loaded axially in tension and compression, loaded in torsion, and subjected to both internal and external pressure; all within the elastic range. Nine elastic constants were determined in this manner. Young's moduli and Poisson's ratios in the longitudinal direction were established for both tension and compression by the axial loading tests. Poisson's ratios and Young's moduli in the hoop direction were obtained from internal pressure tests for tension, and from external pressure tests for compression. Shear moduli were determined from torsion tests.

After each specimen is subjected to all elastic loading conditions, post elastic behavior will be investigated to failure in axial tension, axial compression, and external pressure.

Test results. The cylinder tests are currently in progress. Preliminary values of the nine elastic constants based on results of only one cylinder of each type are illustrated in figure 14. For each elastic constant, the center bar represents the theoretical value; the upper bar, the experimental tensile value; and the lower bar, the compressive value. The theoretical values are generated from a rather complex mechanical analysis which takes into account the properties of both the matrix and reinforcement, and the interactions between the two throughout a multilayer, multidirectional filament wound cylinder (ref. 6). The theoretical values are calculated for a measured fiber volume fraction of 40 percent for the glass cylinders and 42 percent for the boron cylinders. These values were determined by either leachout or burnout tests of sections removed from the cylinders after failure.

The extent of agreement between experimental and calculated values of the elastic constants is reasonably good considering the preliminary nature of the experimental data. Experimental moduli values averaged about 115 percent of theoretical values for glass, and about 125 percent of theoretical for boron. Although the results to date are not complete, it seems possible that present theory used to predict elastic constant values for glass reinforced cylinders may be equally satisfactory for boron specimens.

The calculated weights of a series of cylinders designed to resist axial compression, external pressure, and internal pressure loads are shown in figure 15. The experimental elastic constants determined from the tests of the 3-inch-diameter by 6-inch-long cylinders, and the composite tensile strengths determined from the ring specimens were used to design boron or glass reinforced cylinders 30 inches in diameter by 54 inches long. The purpose of figure 15 is to compare these hypothetical boron and glass cylinders on the basis of total cylinder weight for each loading condition. The first two loading conditions result in compression of the reinforcing fibers and, accordingly, the hypothetical cylinder design was based on the use of the isotropic winding pattern. The internal pressure load places the fibers in tension. The design for the cylinders to resist internal pressure was based on the orthotropic wrapping pattern. The 600,000-pound load applied to the hypothetical cylinders in axial compression is the maximum load at which both the boron and glass cylinders fail by buckling. For cylinders of these proportions this is approximately the limiting load for the buckling failure mode; higher loads could be attained by increasing the wall thickness but would result in failure due to splitting or delamination of the composite wall of the cylinder. Similarly, the 1000 lb/sq in.

external pressure design load is the greatest pressure at which both cylinders will undergo buckling failure. The 1000 lb/sq in. value for the internal pressure design load was chosen to emphasize the difference in structural requirements between cylinders designed to withstand external and internal pressure. Based on the calculations, it appears that the boron reinforced cylinders exhibit 45-percent and 35-percent weight advantages in axial compression and external pressure, respectively, due to the much greater stiffness of the boron filaments. In the case of internal pressure loading, however, the caluclations indicate that the boron cylinder is at a disadvantage because failure is governed by the composite tensile strength. The significantly higher tensile strength of the glass reinforced cylinder gives it a 60-percent weight advantage over the boron cylinder.

4. CONCLUDING REMARKS

On the basis of a comprehensive study of single filament mechanical properties, boron filaments are characterized by moderate strength and high stiffness when compared with filaments of glass. The boron filaments involved in this study had no appreciable strength at 1750° F.

Results of tensile tests on boron-epoxy composites showed that the ratio of composite fiber stress at failure to virgin fiber strength was significantly less than that for similar glass reinforced composites.

The composite structure test program is not complete. The elastic constants presented herein result from tests of 4 of the 14 specimens to be tested. However, on the basis of this preliminary data, it seems apparent that present theory used to predict elastic constant values for glass reinforced cylinders may be equally satisfactory for boron specimens.

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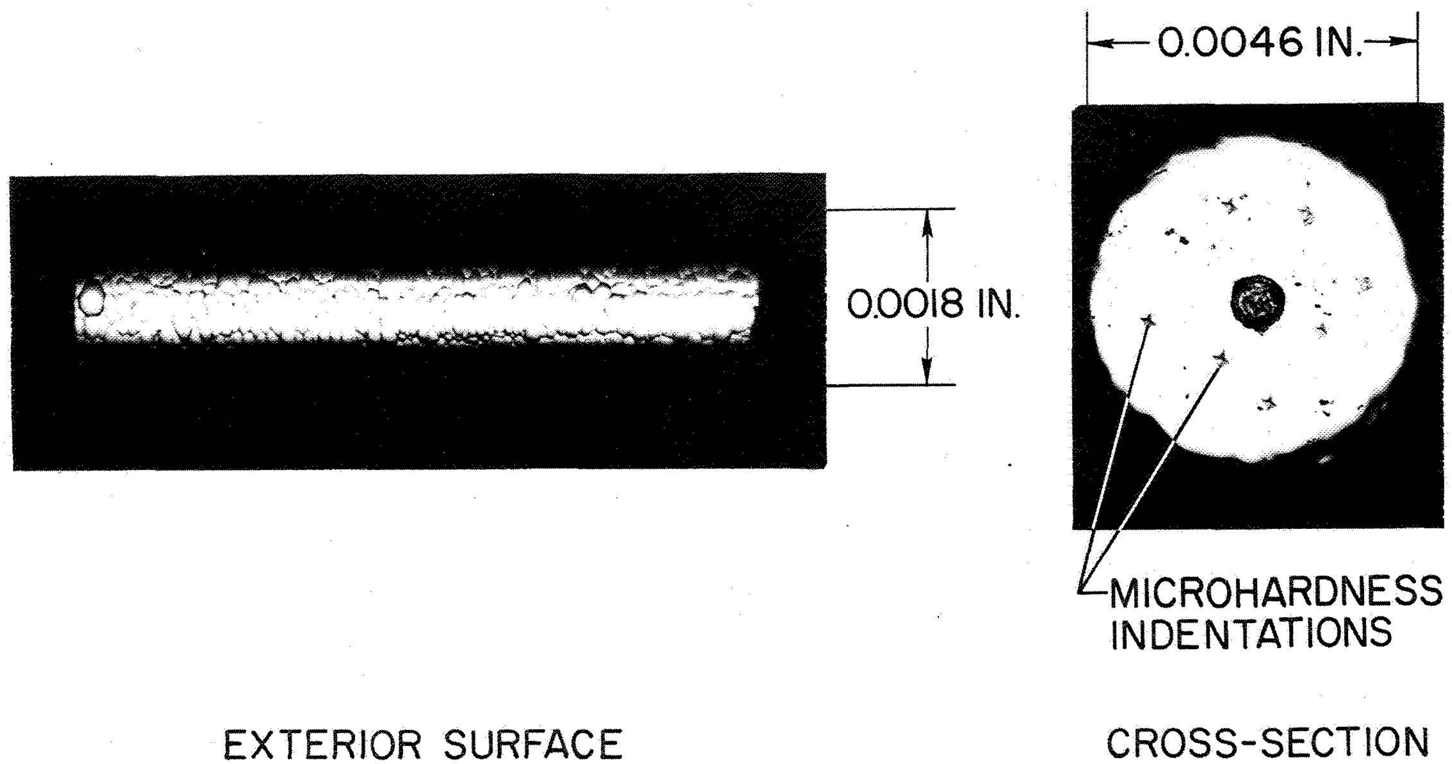

Figure 1.- Boron filaments.

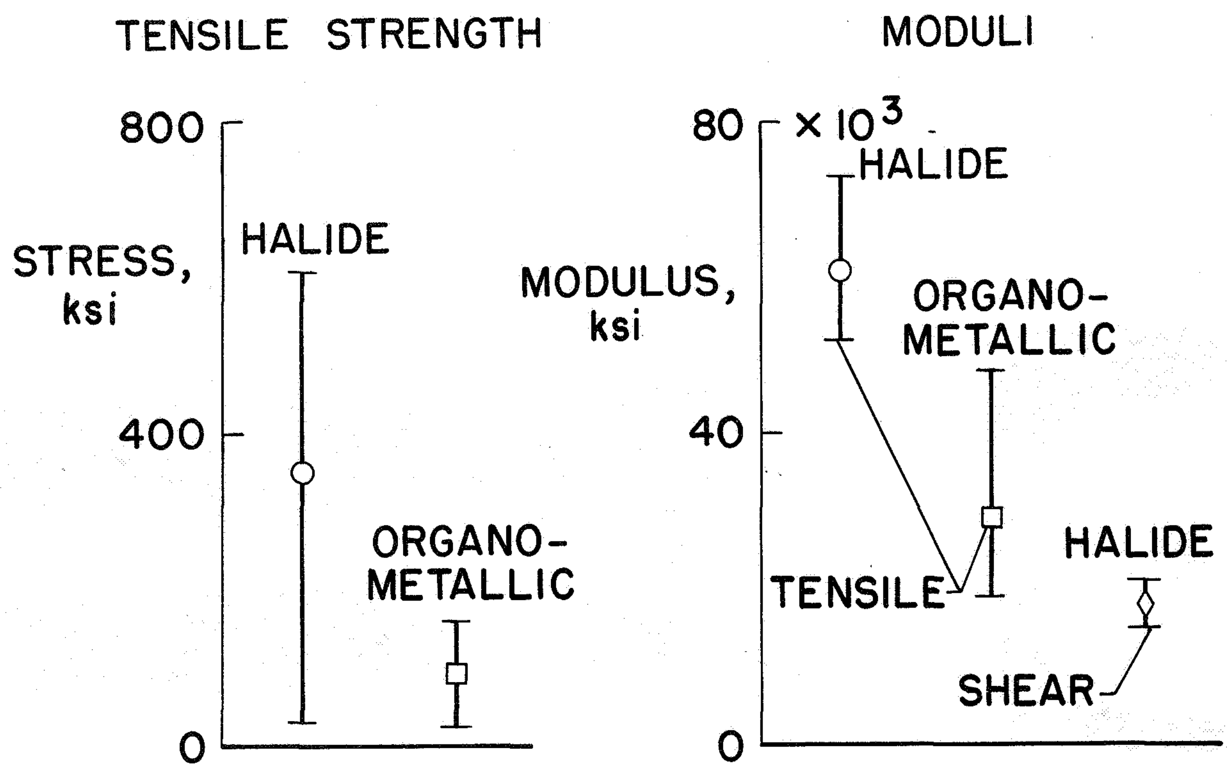

Figure 2.- Mechanical properties of boron filaments.

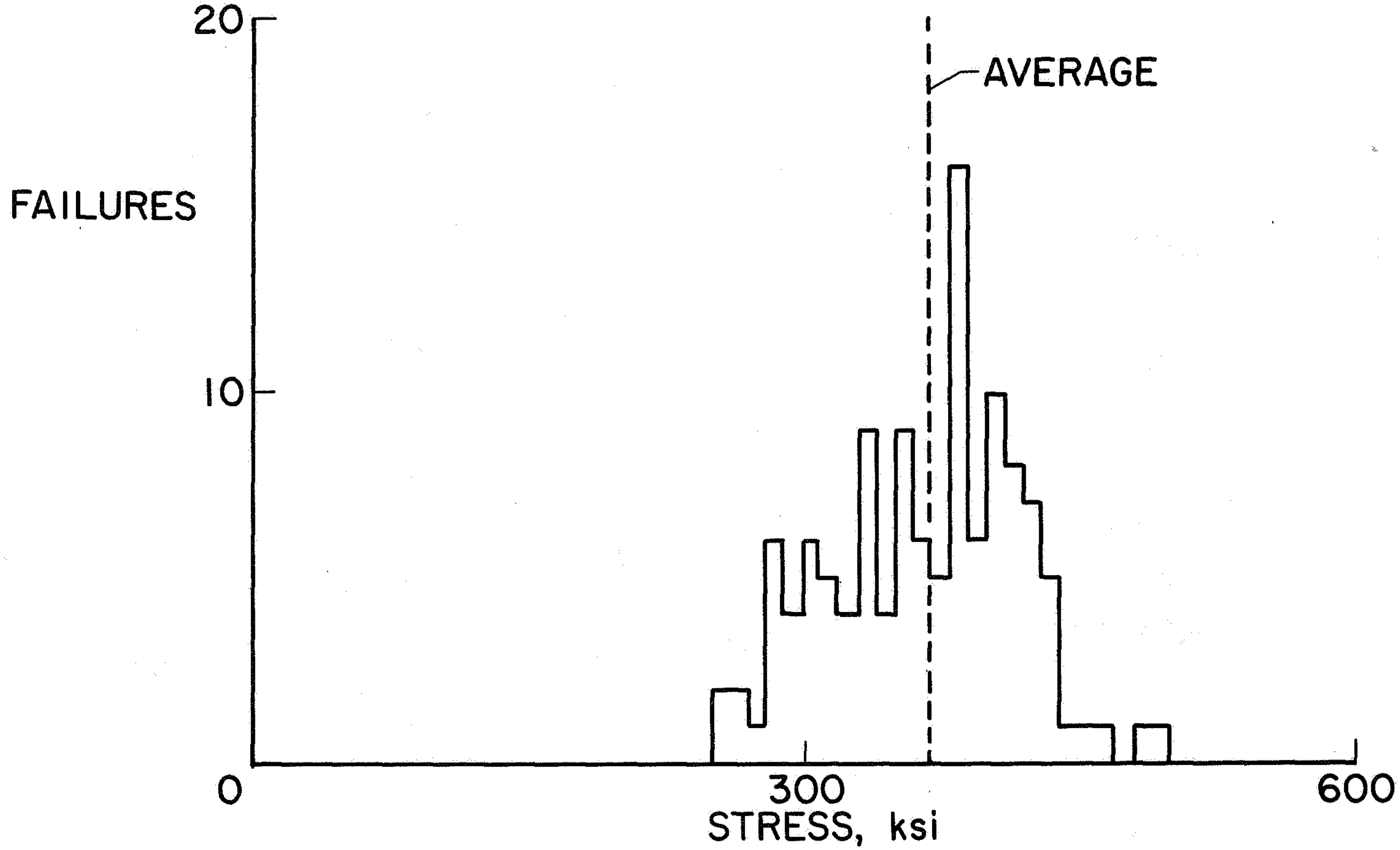

Figure 3.- Distribution of halide filament tensile failures.

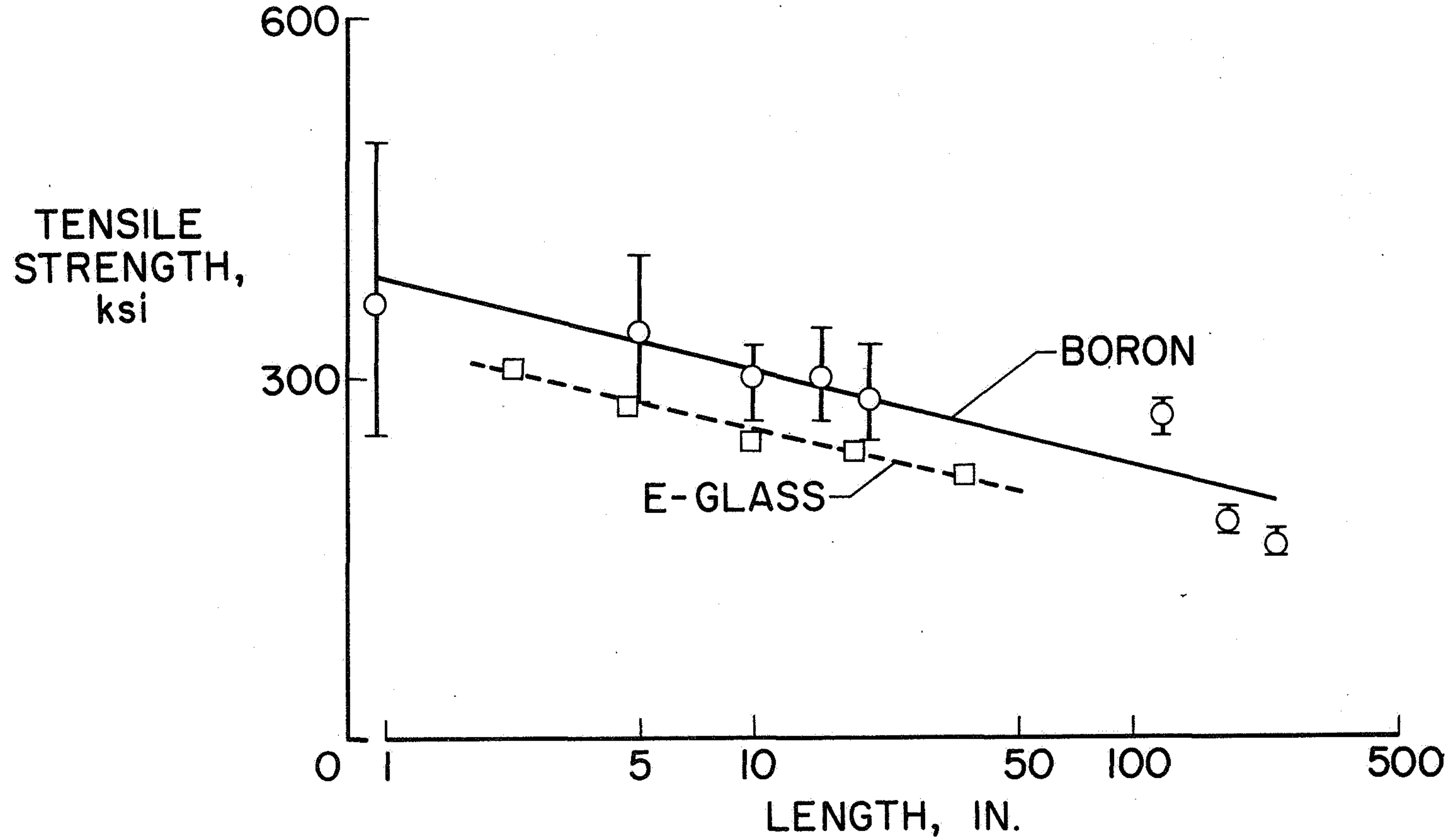

Figure 4.- Effect of length on strength.

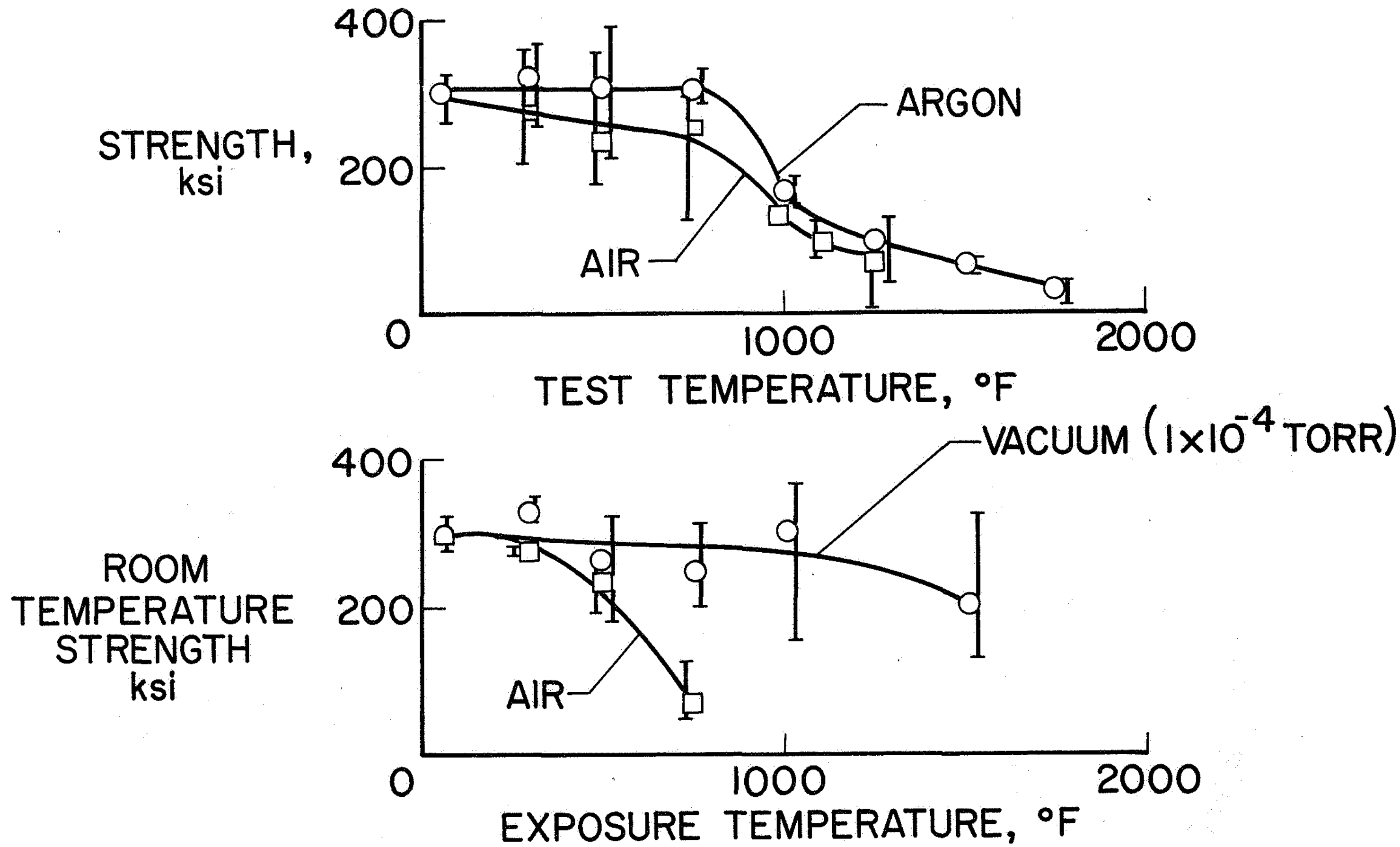

Figure 5.- Effects of temperature on tensile strength of halide filaments.

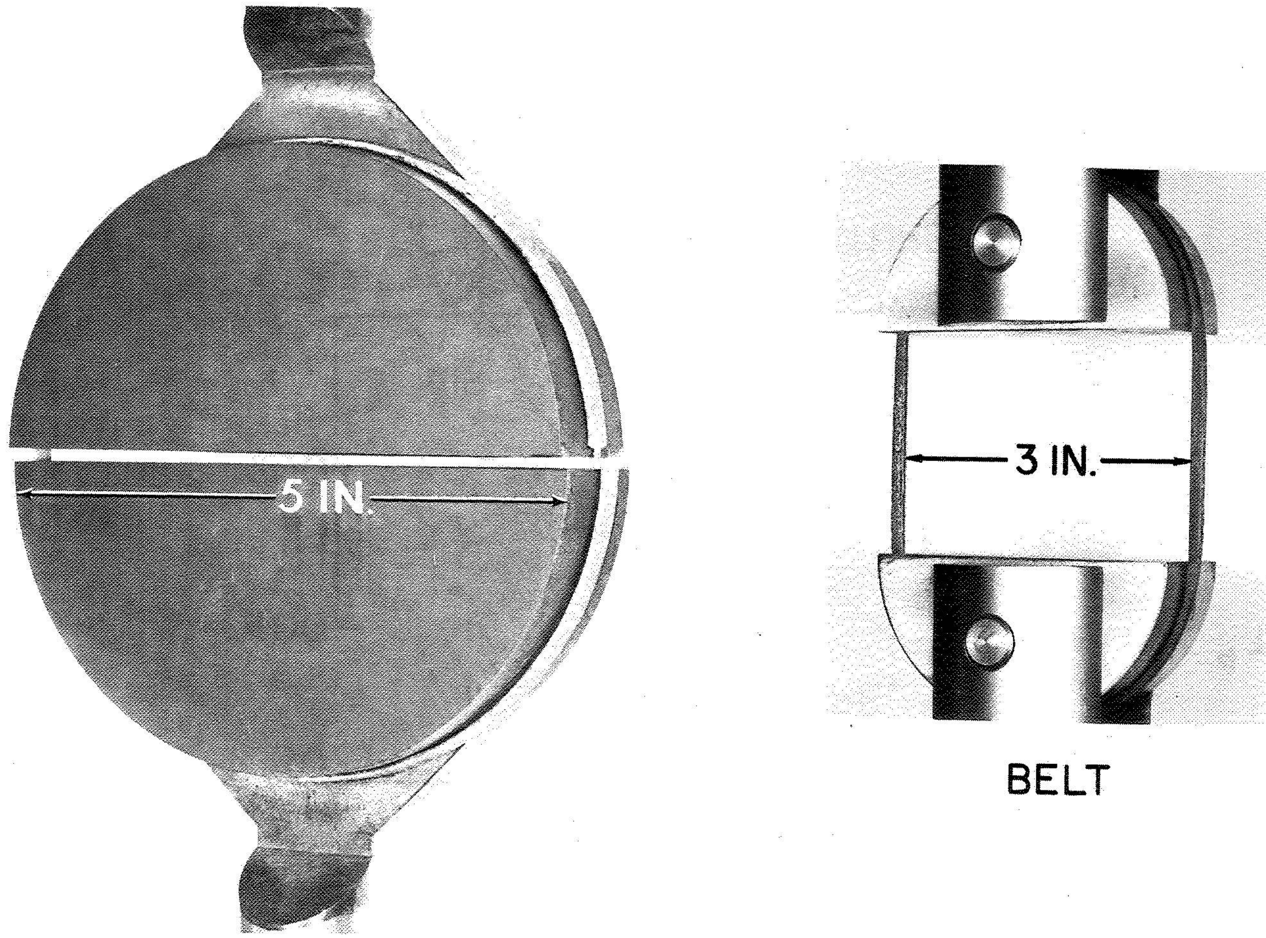

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Figure 6.- Composite specimens and test fixtures.

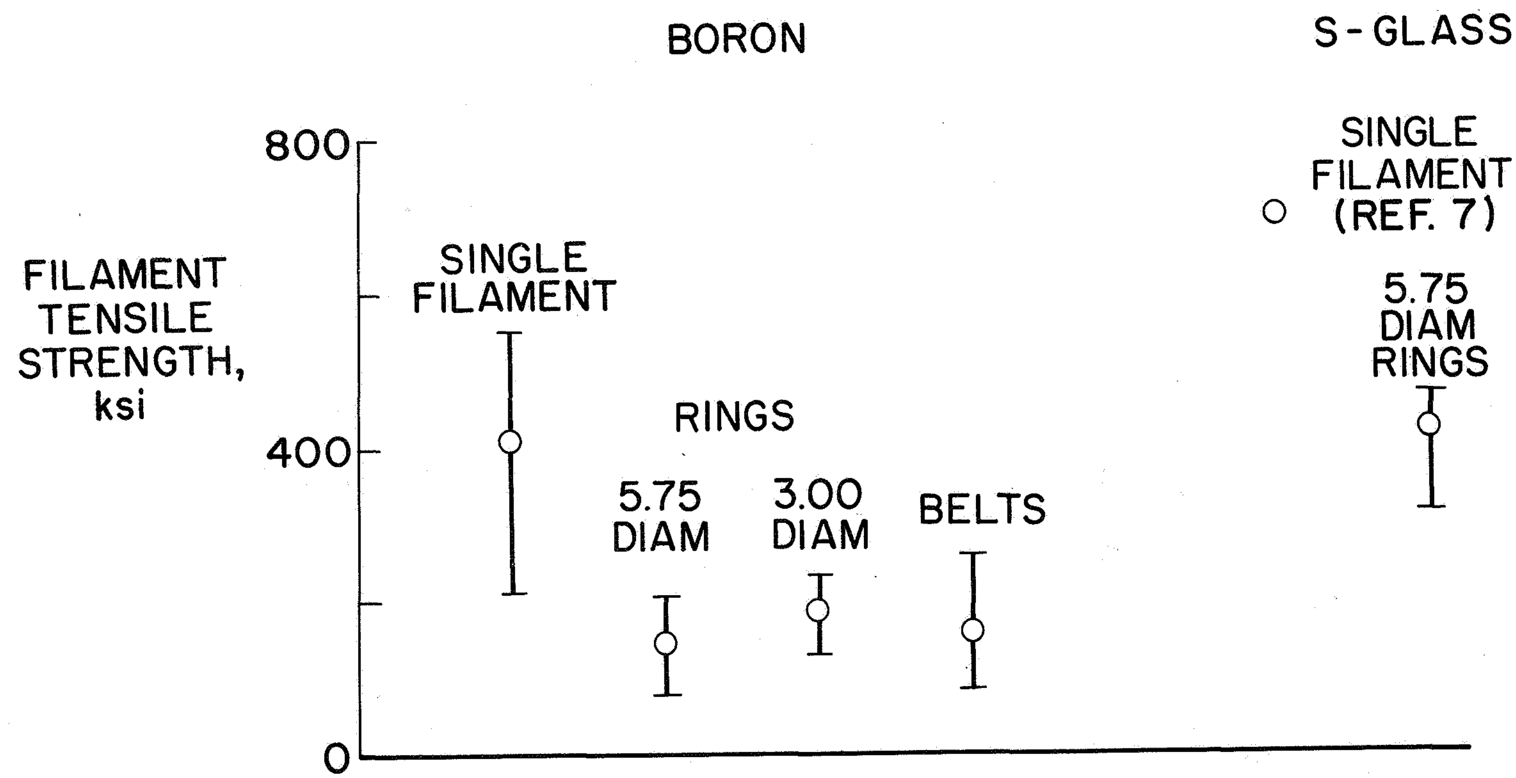

Figure 7.- Comparison of composite and single filament strengths.

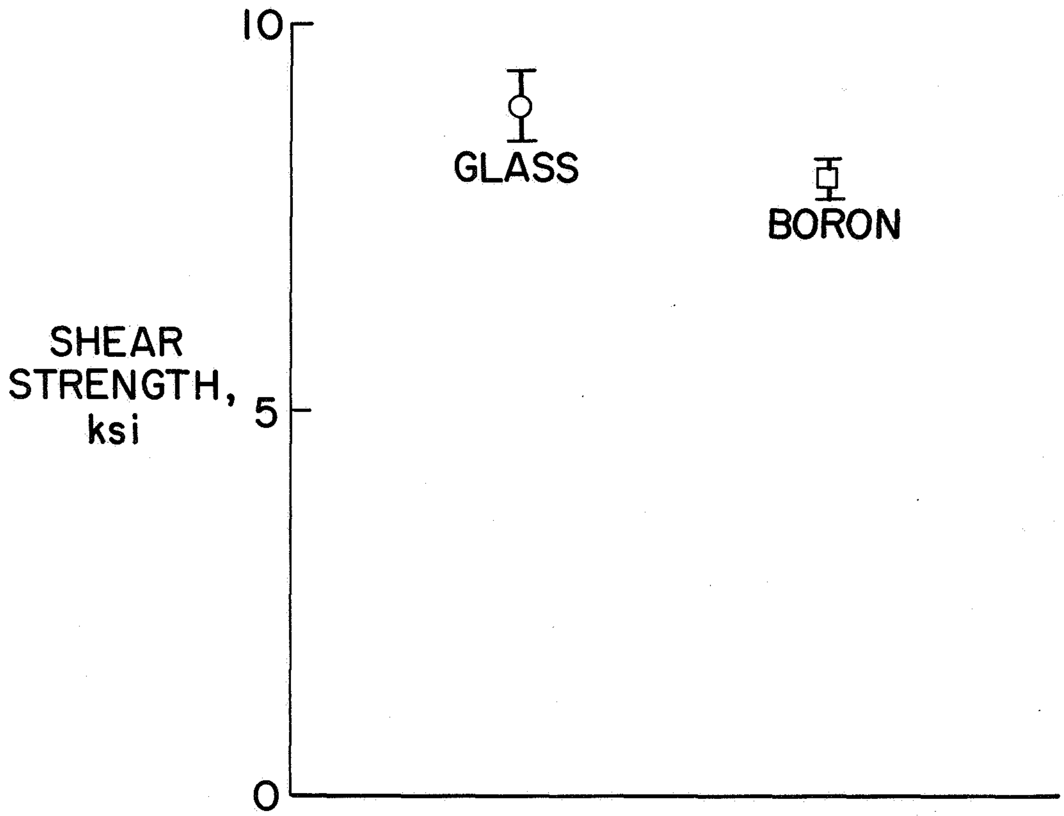

Figure 8.- Interlaminar shear strength.

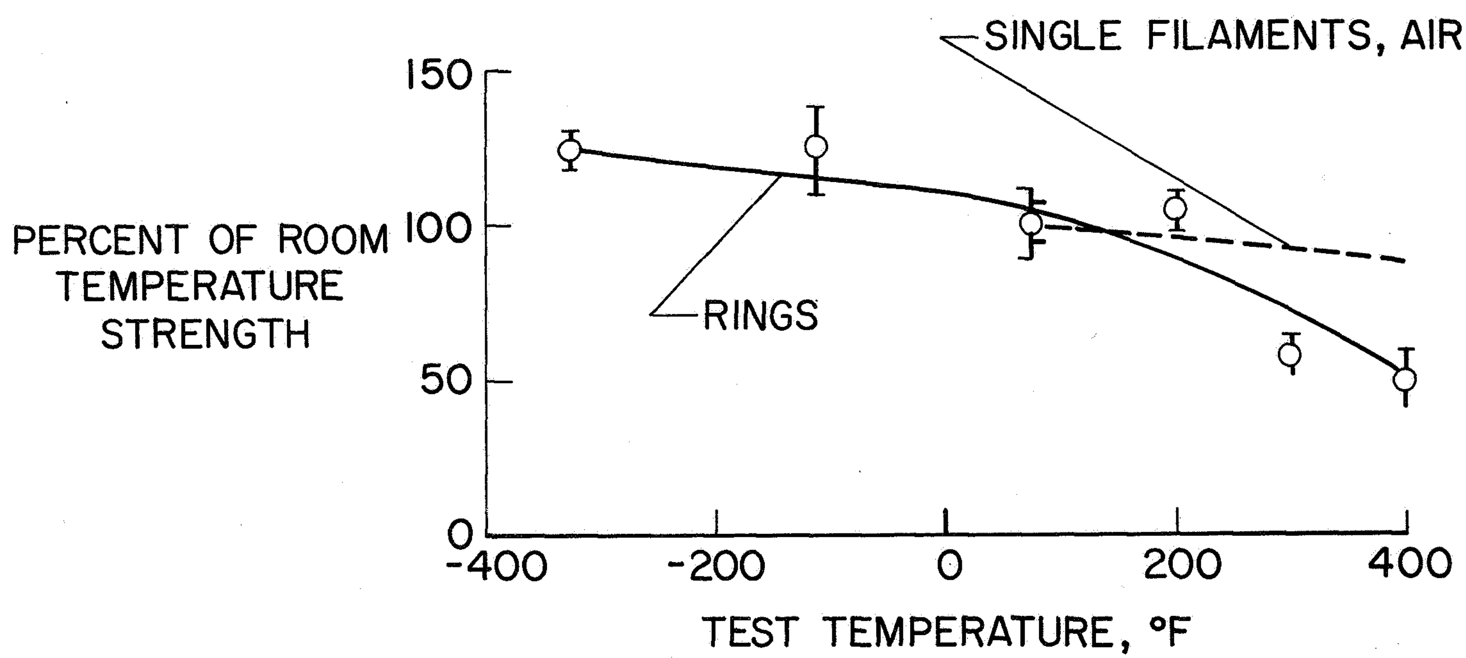

Figure 9.- Effect of temperature on boron composite strength.

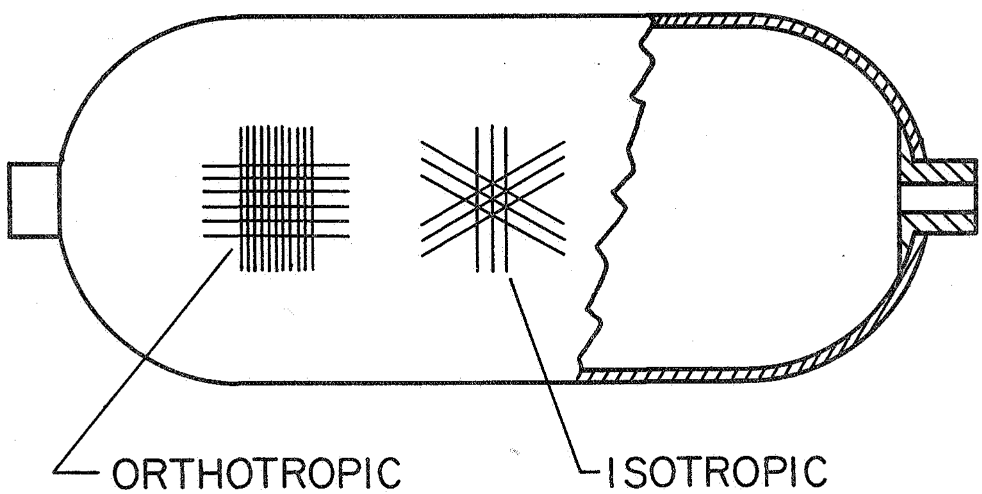

Figure 10.- Bottle winding patterns.

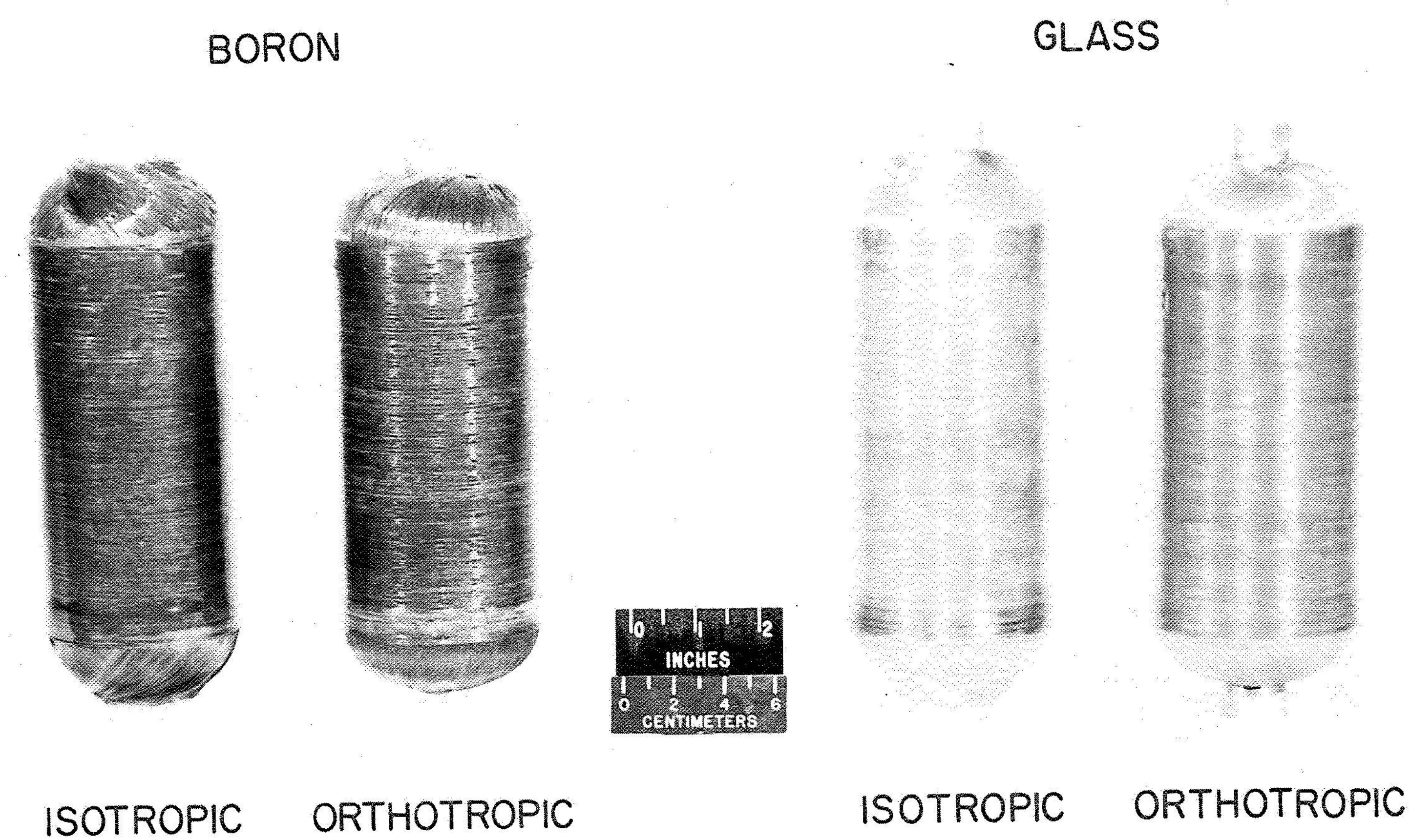

Figure 11.- Boron and glass bottles.

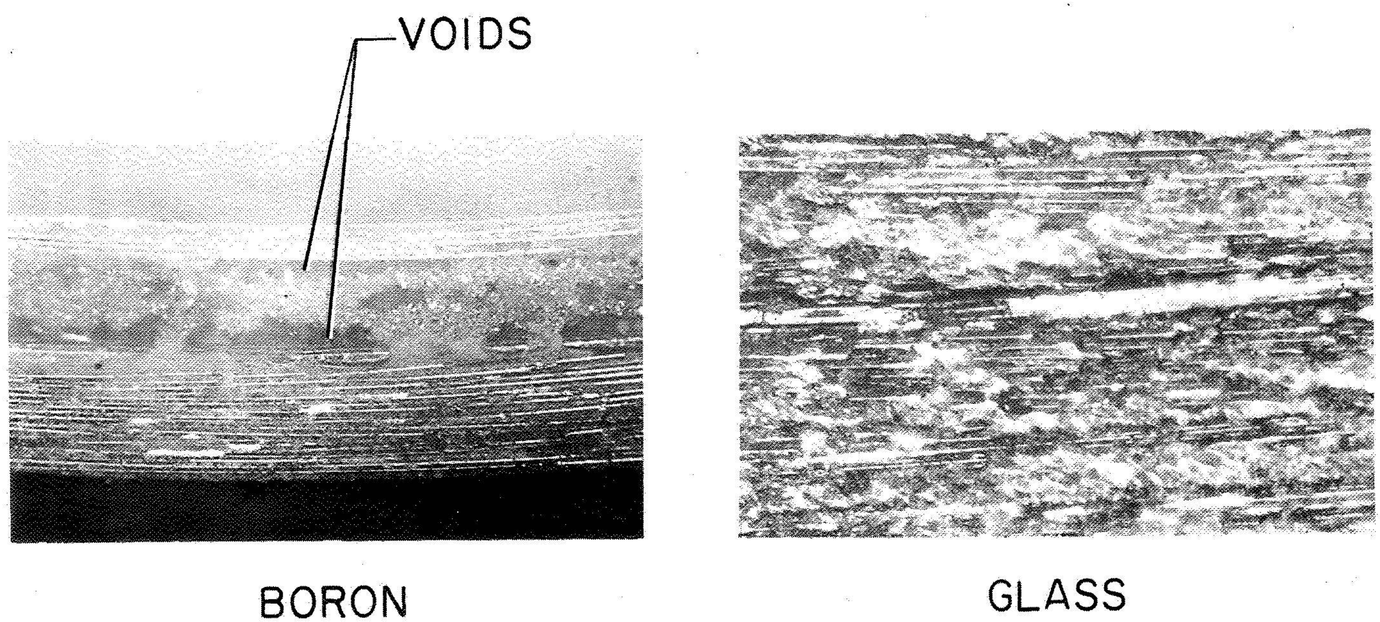

Figure 12.- Cross sections of cylinder walls.

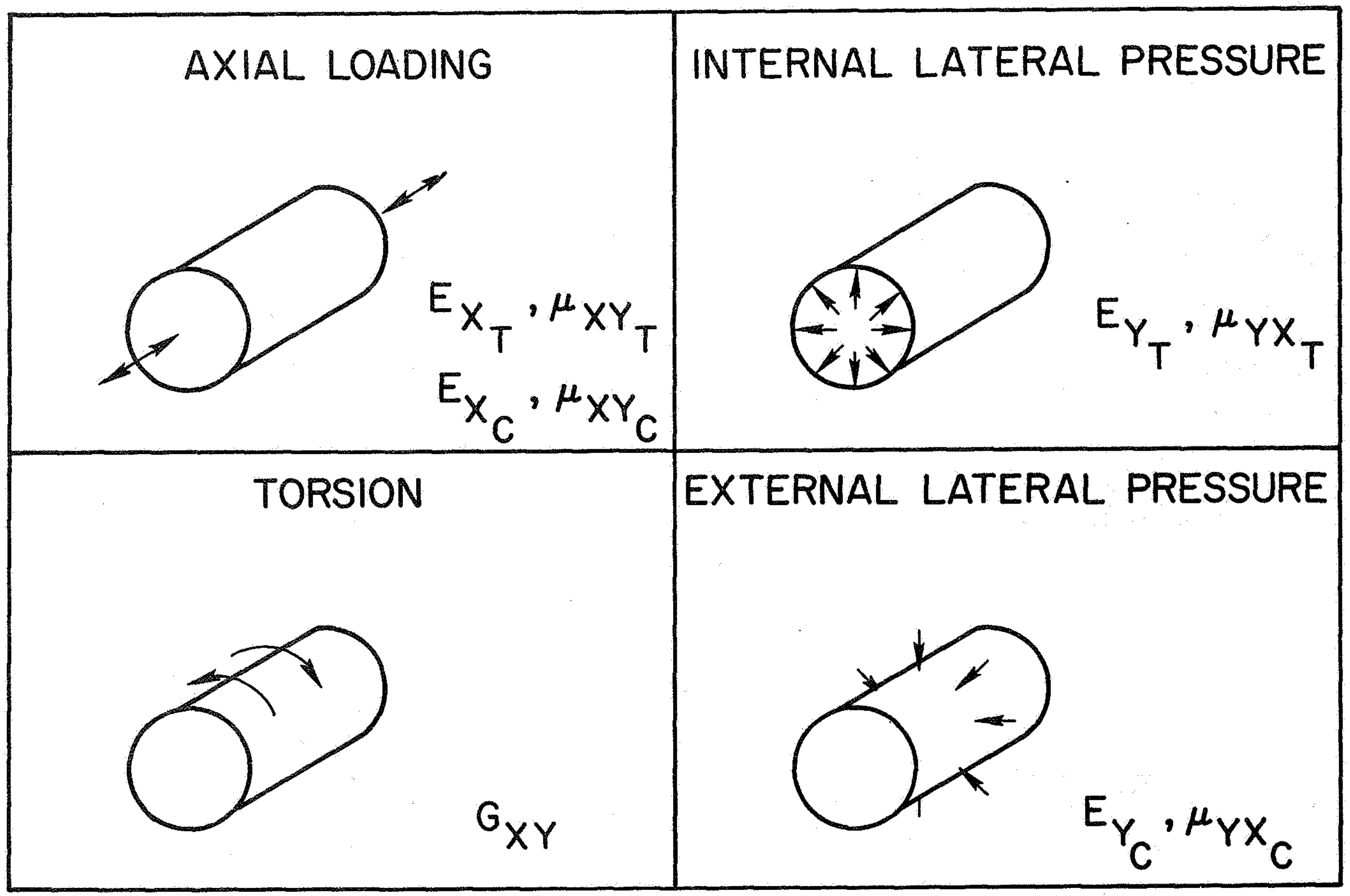

Figure 13.- Cylinder tests and objectives.

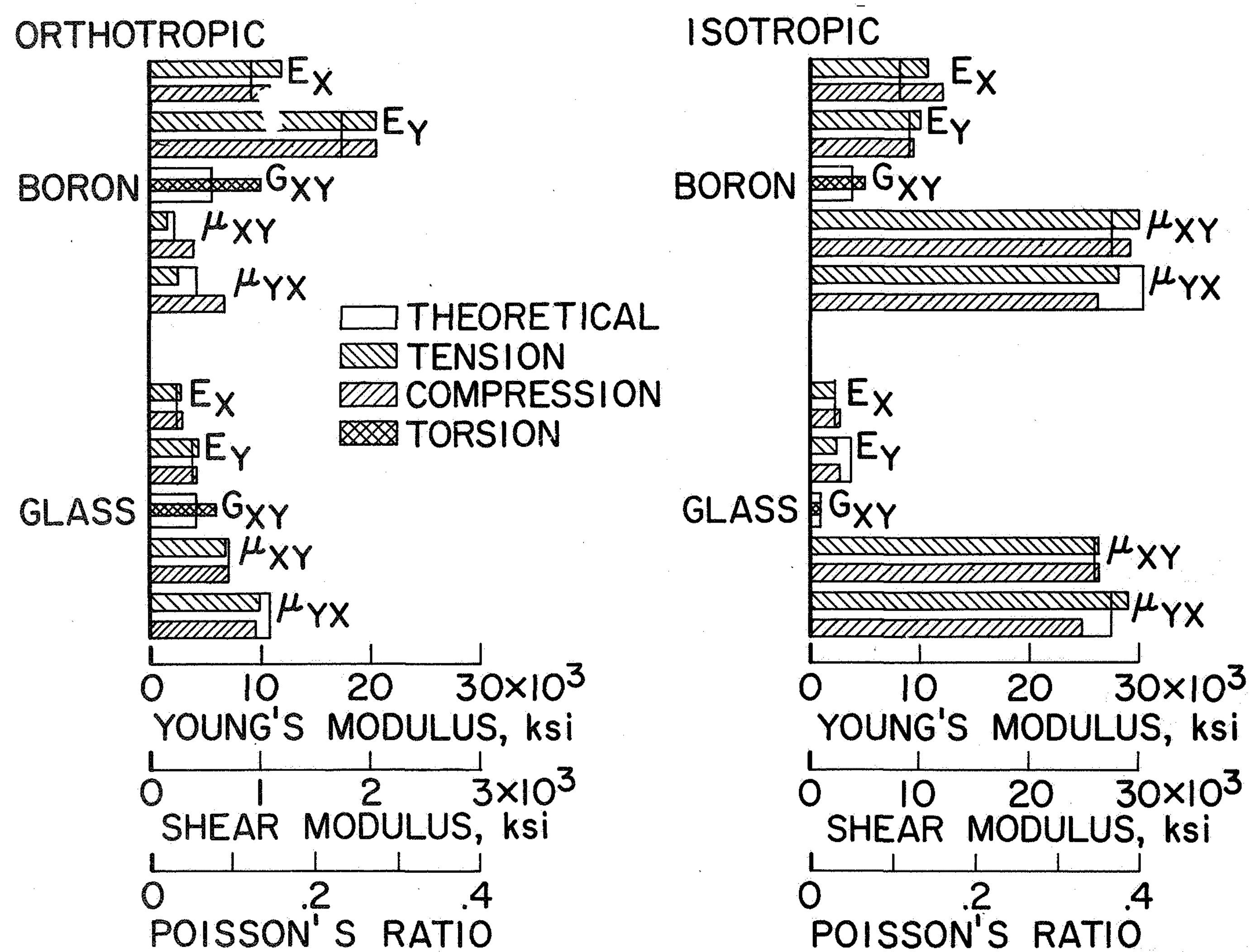

Figure 14.- Comparison of experimental and calculated elastic constants.

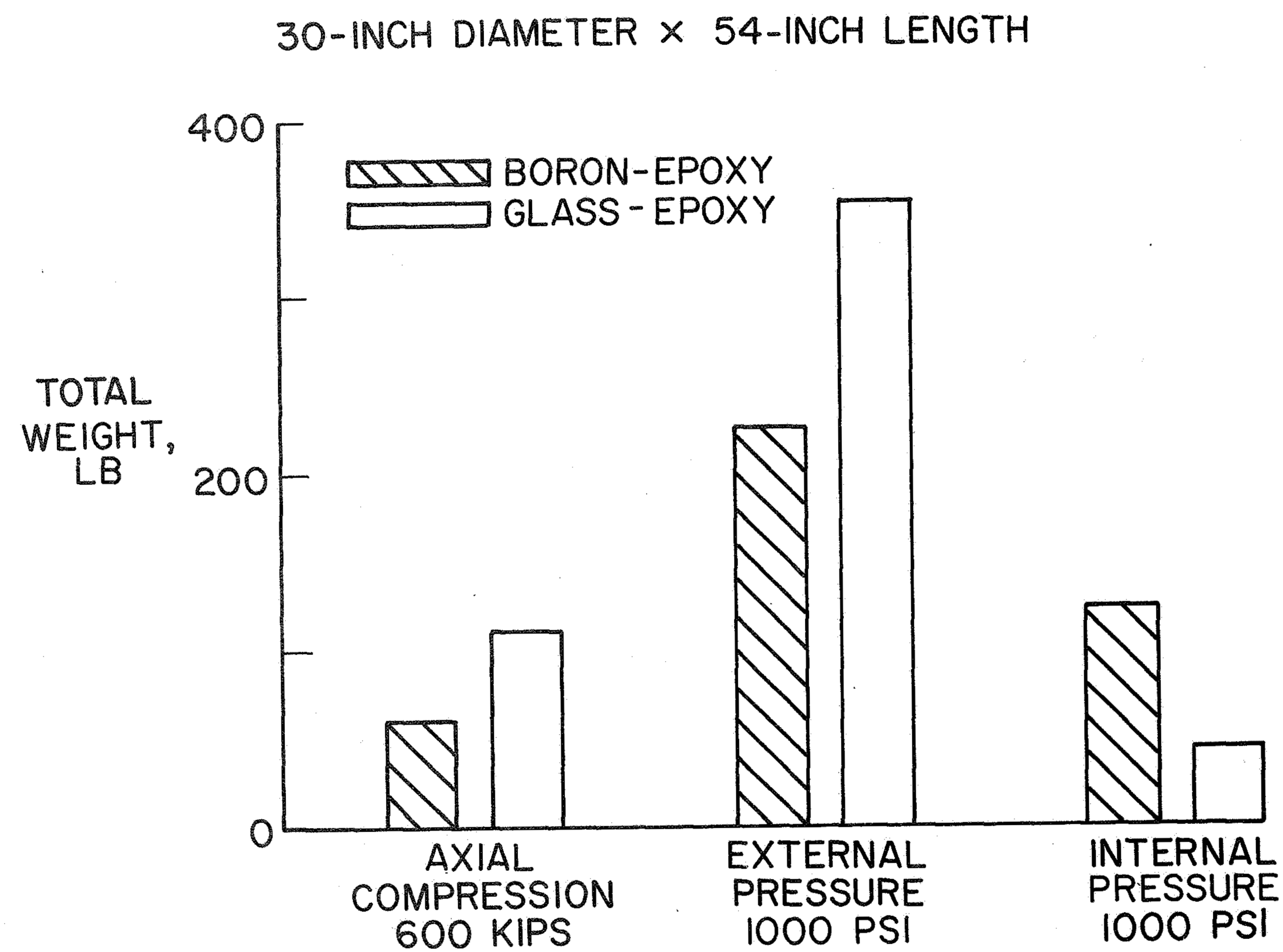

Figure 15.- Cylinder weight comparison.

NASA-Langley, 1966